Establishing National Traceability and International Comparability

Technical Highlights of the Analytical Chemistry Division of NIST

Definitions . . .

- **Reference Material (RM):**

  A material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. *[ISO VIM: 1993, 6.13]*

- **Certified Reference Material (CRM):**

  A reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence. [ISO VIM: 1993, 6.14]

- **NIST Standard Reference Material® (SRM®):**

  A CRM issued by NIST **that also meets additional NIST certification criteria.**

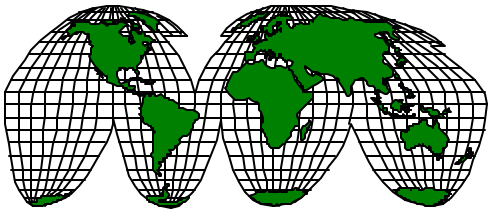
- **NIST Traceable Reference Material™ (NTRM™):**

  A commercially produced reference material with a well-defined traceability **linkage to existing NIST standards for chemical measurements**. This traceability linkage is established via criteria and protocols defined by NIST to meet the needs of the metrological community to be served.

Traceability: The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, all having stated uncertainties.

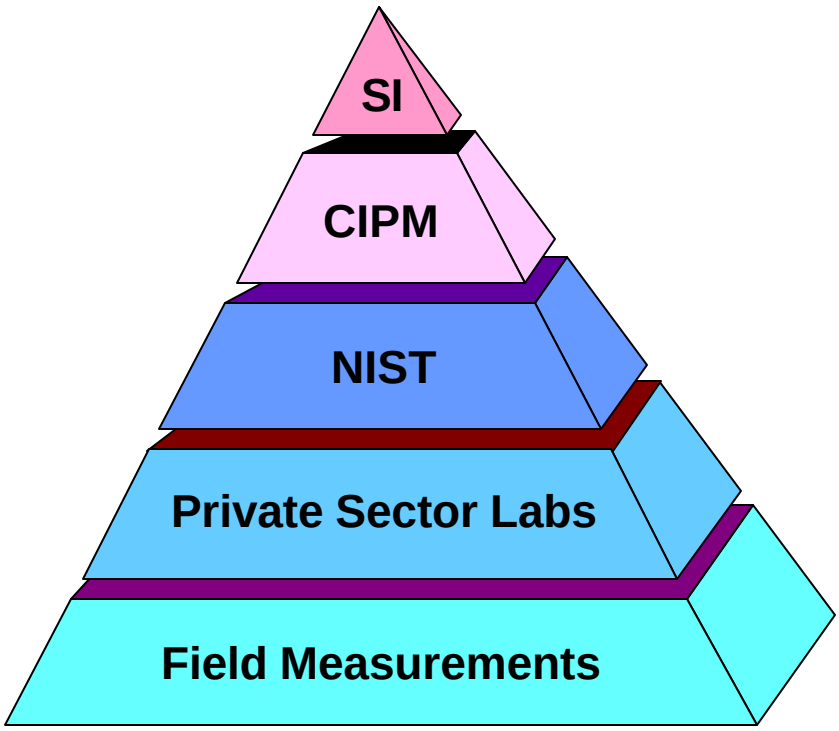
[**VIM**: **I**nternational **V**ocabulary of basic and general standard terms in **M**etrology, ISO, Geneva, Switzerland, 1993, 6.10]

Traceability of Chemical Measurements

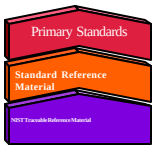


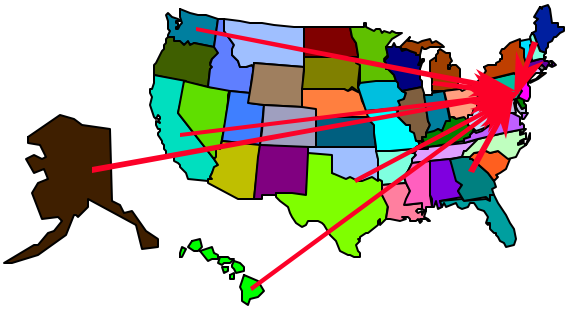
Important for:

- **Trade**
- **Healthcare Decision-Making**
- **Environmental Decision-Making**
- **Global Science**



NIST Traceable Reference Materials (NTRMs)





Certified Reference Materials:

- CRMs are critical for establishing the unbroken chain to national standards (NIST).

However:

- NIST does not have the resources to provide the quantities and specific varieties needed for all the measurements made in the U.S.
  - exact matrices
  - unique compound combinations
  - concentrations
  - etc.

Therefore:

- NIST is working with private sector reference materials producers to develop **N**IST **T**raceable **R**eference **M**aterials **(**NTRMs).
  - **Existing:**
    - Gas NTRMs
  - **Under Development:**
    - Optical filter standards
    - Elemental solution standards
    - Organic solution standards
    - Metal alloy standards

Why NTRMs?

- Faster Response to User Needs
- Better Availability of NIST Traceable Materials
- Better Leverage and More Appropriate Use of NIST Resources

⎨ Reallocation of NIST Resources to Focus on New Measurement Problems

NIST Traceable Proficiency Testing (PT) Studies For EPA/States Water Programs

Since the 1970's, EPA has conducted semiannual proficiency testing (PT) to assess the competence of over 5,000 public and private sector laboratories to conduct analyses required by the Clean Water and the Safe Drinking Water Acts. At the end of 1998, the cost-free provision of these services will be phased out and replaced by a multiprovider system in which interested states and private companies will provide these PT services on a fee-basis. Mechanisms and tools to provide appropriate government oversight of these programs are needed.

EPA will -

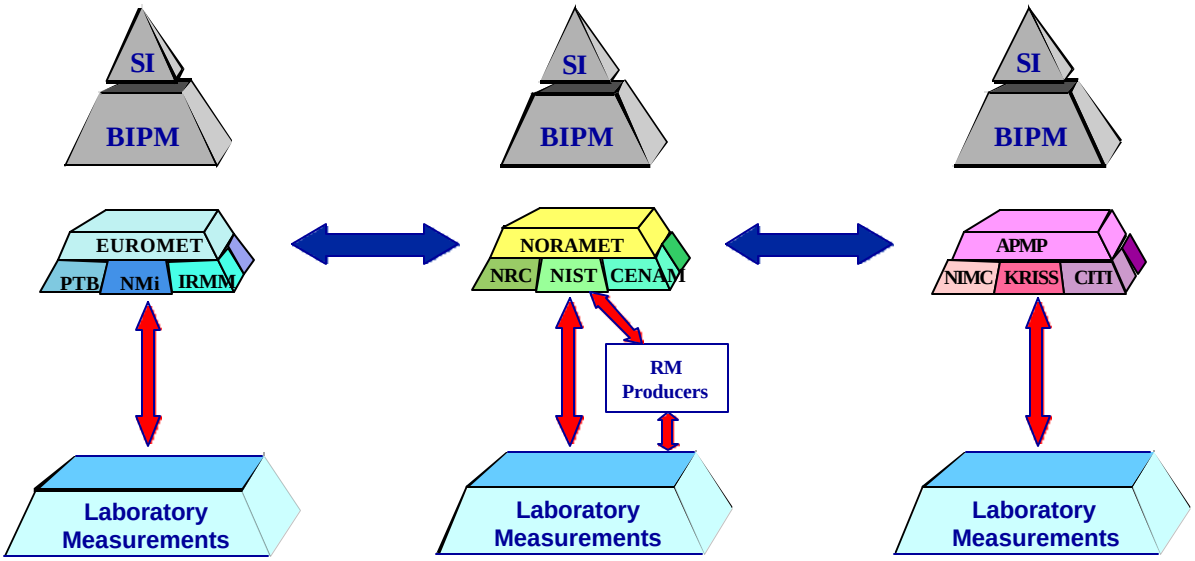
- provide NIST with 3-y support to develop a program for private sector/state provision of PT studies
- work with States and NIST to assure that program for (1) preparing, value-assigning, and distributing PE samples and (2) evaluating and reporting Environmental Testing Laboratory PE data is sufficient to support the needs of the Nation's environmental water programs.

NIST will -

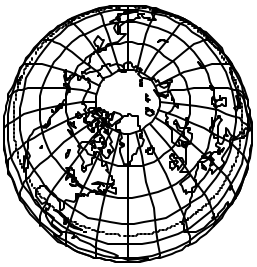
- develop and manage program for accrediting private sector PE study providers
- establish and maintain Primary Benchmark Materials to support the program
- conduct blind sample audits of the commercially supplied PT samples on an ongoing basis as part of our QA responsibility for the program

PE STUDY PROVIDERS will -

- develop, manufacture, value-assign, and distribute PE Study Samples
- score results and report data to EPA, NIST and the States
- maintain accreditation through NIST NVLAP



Horizontal Chemical Measurements Comparability Activities



- NORAMET (North American Metrology)
  - US, Canada, Mexico
- SIM (Interamerican Metrology System)
  - The Americas
- CCQM (CIPM Consultative Committee on Amount of Substance)
- Strategic Collaborations with National Metrology Laboratories

Intercomparison of NIST Primary Methods and Standards with Other National Metrology Laboratories

Project Description: To assess and document international comparability for chemical measurements among the world's national chemical metrology laboratories and to link such comparability to the U.S. and North American systems of chemical measurement traceabiltiy for improved accuracy in chemical measurements.

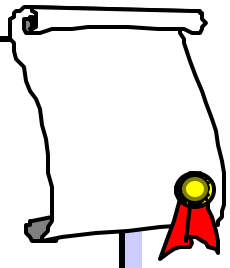
Results: NIST is expanding efforts to document the comparability of NIST primary methods and standards with those of other nations and metrological regions through both multilateral (CIPM CCQM) and strategic bi- and multi-lateral collaborations.

Strategic collaborations with National Metrology Laboratories for assessing the equivalence of primary standards include:

–NMi	*auto emissions gases*
–DFM and OHM	*conductivity*
–PTB	*pH*
–NIMC and BAM	*elemental solution standards*
–NIMC	*volatile organic compound standards*

Our bilateral program with NMi (The Netherlands) has resulted in a formal "Declaration of Equivalence" that is mutually recognized by the U.S. EPA and European environmental regulatory bodies as documenting the equivalence of seven NIST and NMi primary gas mixtures suites (spanning a wide range of concentrations).

Declaration of Equivalence



DECLARATION OF EQUIVALENCE

The National Institute of Standards and Technology - NIST
Gaithersburg, MD, United States of America

and

The Netherlands Measurements Institute - NMi
Delft, The Netherlands

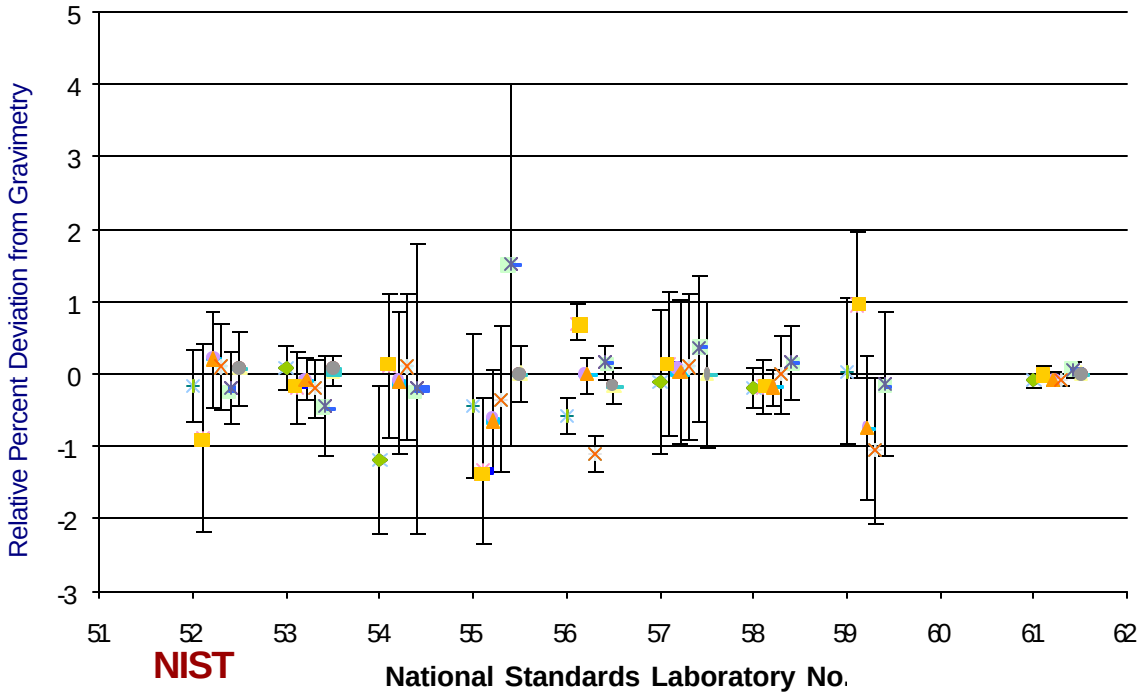
NIST and NMi declare that on July 1, 1997 the suites of primary standard gas mixtures developed and maintained in both the Institutes, comprising a range of analyte concentrations in the stated diluent gas as listed in Annex 1, can be considered as equivalent within the stated uncertainties. This declaration shall expire on July 1, 1998 at which time a new declaration shall take effect.

This declaration is based on the results of intercomparisons carried out between the two Institutes. A continuous program of intercomparisons has been agreed to in order to maintain this declaration and is outlined in a mutual Memorandum of Understanding, effective, July 1, 1994.

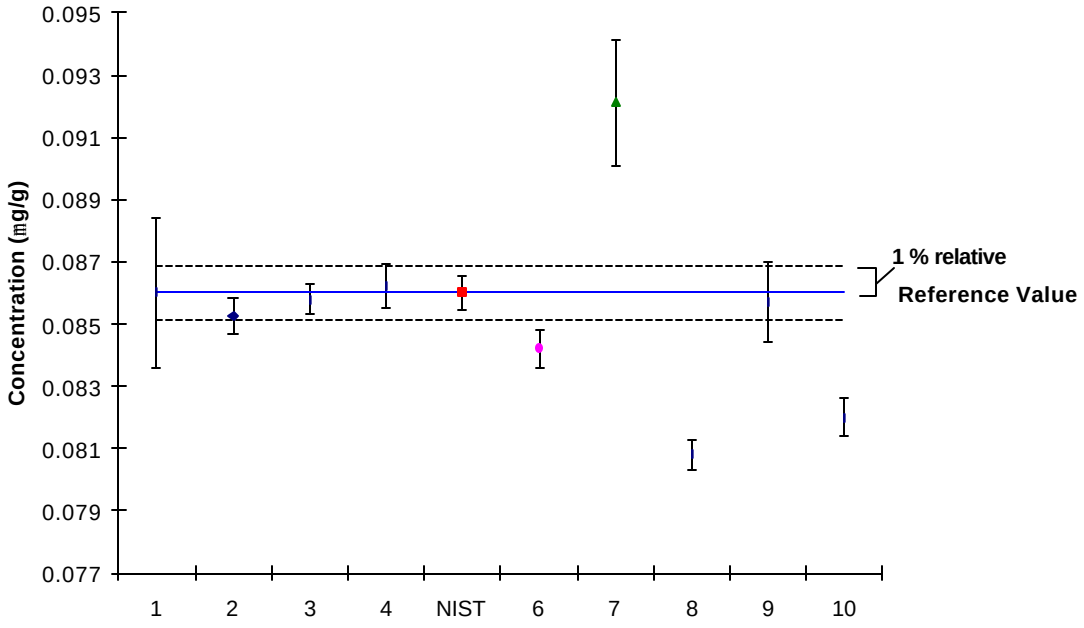
Annex 1: NMi and NIST Primary Standard Gas Mixture Suites which are declared to be equivalent

Component in N_2	Molar Fractions (mol/mol)	Average Bias (% relative) NIST-NMi	Statistical Test for Statement of Equivalence	Date of Reassessment
Carbon Dioxide	$1.5*10^{-1}$ to $1*10^{-4}$	0.07 %	Bias < 0.5 %	1996
Carbon Monoxide	$6*10^{-2}$ to $1*10^{-4}$	0.09 %	Bias < 0.5 %	1996
Ethanol	$2.5*10^{-4}$ to $1*10^{-4}$	0.9 %	Bias < 1 %	1998
Oxygen	$2.2*10^{-1}$ to $2*10^{-2}$	0.05 %	Bias < 0.5 %	1998
Propane	$3*10^{-3}$ to $5*10^{-4}$	0.08 %	Bias < 0.5 %	1996
Nitric Oxide	$1*10^{-5}$ to $4*10^{-3}$	0.2 %	Bias < 0.5 %	1998
Sulfur Dioxide	$1*10^{-4}$ to $3.5*10^{-3}$	0.05 %	Bias < 1 %	1998

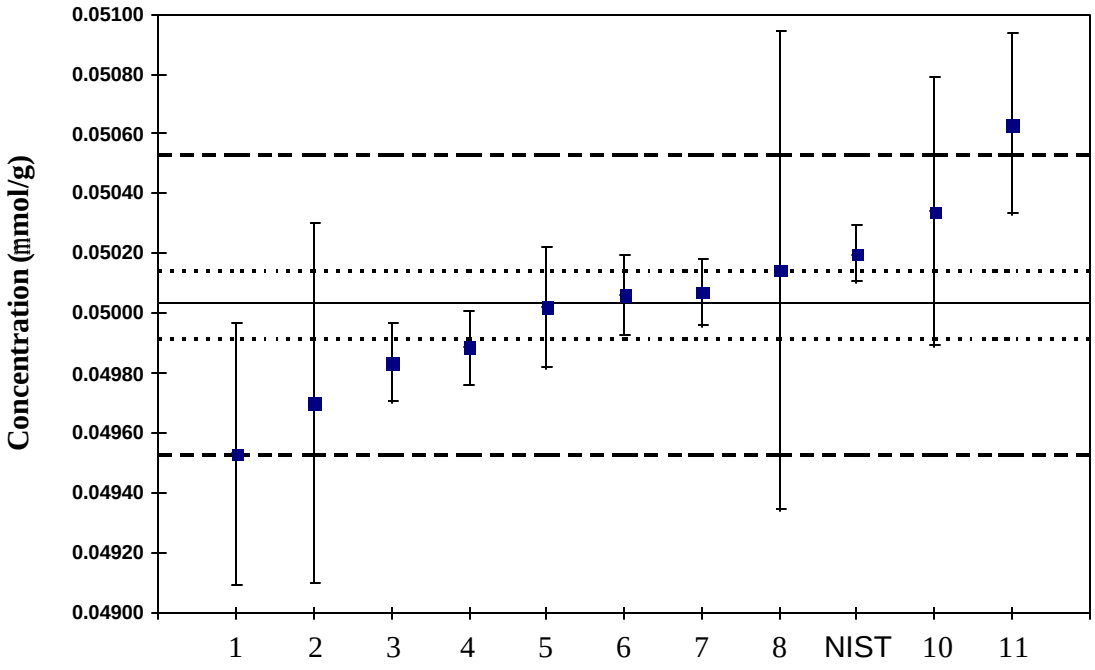
FY97 CCQM Study - Natural Gas



First CCQM Study on the Determination of 4,4'-DDE in "Isooctane" - Solution 1 ('96)



Third CCQM Study - Results for Pb in Water (‘96)



National Standards Laboratory No.